DESIGN OF A CENTRIFUGE ROTOR CAP (A)

Mr. Ken Jacobsen, project engineer at the Spinco Division of Beckman Instruments Inc., had proposed to incorporate new features in an ultracentrifuge. These features had been developed in a national laboratory. They now had to be improved and refined for the Spinco production model. Some background on the operation of ultracentrifuges is given; attention is focused on the detail design of a subsystem and on the technical difficulties which Mr. Jacobsen had to surmount to achieve a satisfactory product.

Prepared in the Design Division, Department of Mechanical Engineering by Prem C. Garg under the direction of Professor H. O. Fuchs with financial support from the National Science Foundation. The cooperation of Mr. Ken Jacobsen of the Spinco Division of Beckman Instruments Inc. is gratefully acknowledged.

Separation of a complex structure into its cellular and sub-cellular constitutents is one of the most important tools in bio-chemical studies. Though a number of different techniques exist for specific applications, centrifugal separation is the one most commonly used for this purpose. Rapid developments in bio-chemistry and micro-biology have resulted in a continuous demand for 'higher resolution' centrifuges capable of handling large sample quantities. Present day research centrifuges run at over 60,000 RPM (as compared to about 3,000 RPM for centrifuges used in college chemistry laboratories) and are capable of giving accelerations in excess of 400,000 g.

Spinco Division of Beckman Instruments in Palo Alto, California, specializes in sophisticated instrumentation for bio-chemical and physiological research and is the major supplier of ultracentrifuges used in research. Over the years the company has regularly improved its products, mostly through its own development programs and occasionally by adapting from the work done by outside research workers.

Depending upon the physical properties of the particles to be separated, either of the following two methods may be used to accomplish the centrifugal separation.

1. If the particles have different densities they will band at different radial locations in a radial density gradient field,* when rotated for sufficient time.
2. If the particles have different sedimentation rates** they will temporarily band at different locations in a centrifugal field during early phases of rotation, and if the rotation is

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* A liquid whose density varies from the inner radius to the outer radius. Density variations can be obtained by varying the concentration of the solute in the liquid. Sucrose and Cesium chloride are the two commonly used solutes.

** Sedimentation rate of a particle rotating in a centrifuge is the measure of the rate at which the particle approaches its equilibrium position. For a spherical particle it varies as the square of the particle radius as well as the difference in densities of the particle and the liquid gradient at the location of the particle.

stopped before the onset of equilibrium, separation can be achieved even though the particles may have the same densities. Exhibit A-1 shows, schematically, the separation of different sized particles on the basis of their sedimentation rates.

Though the whole process may seem rather simple, conceptually, the actual centrifuging machines are very elaborate systems involving a number of sophisticated subsystems; this can be seen from exhibit A-2 which describes one of the centrifuges marketed by Beckman Instruments. Of all the subsystems, the centrifuge rotor where the actual separation takes place constitutes perhaps the most vital component of the centrifuging systems (the rotor is mounted in the rotor chamber of a centrifuging machine, see item F Exhibit A-2; a few typical rotors are shown in Exhibit A-3). Due to the extremely high centrifugal loading produced at high speeds, the size and speed of a centrifuge rotor are heavily dependent upon the developments in materials technology and stress analysis. It is in the design of the rotors where most innovations take place. In fact, so frequently are new rotors introduced that the rest of the centrifuge is designed with an eye on its potential to adapt to future rotors without major modifications.

Oak Ridge National Laboratory (ORNL), Oak Ridge, Tennessee, operated by the Union Carbide Corporation for the U.S. Atomic Energy Commission, has been doing pioneering work in the field of centrifugal separation. Early in the '60's the Biology Division of ORNL felt that the Swinging-bucket or Fixed-angle rotors, the ones in common use at that time, would not be able to withstand the stringent demands imposed by impending developments in micro-biology. There were two simple reasons for that: first, the existing rotors could handle very small sample quantities only, and secondly, their resolution was limited due to the difficulty of preventing mixing between the separated sample layers during unloading when there was no centrifugal force field. To overcome these limitations, the Biology Division of ORNL, in association with the Technical Division which previously had worked on the problem of isotope

separation, developed the concept of dynamically loaded and unloaded zonal rotors. In such a system a hollow rotor, divided into sector shaped compartments by a core, is used instead of the buckets, and the stabilization of the sample layers is achieved by introducing and removing the liquids while the rotor is running, though at a reduced speed (around 3,000 RPM). Exhibit A-4 explains schematically the working of such a rotor.

After going through a number of development stages ORNL was able to develop a workable system in early 1966. Mr. Ken Jacobsen, Engineer-in-charge of the centrifuge program at Beckman had been following these developments by keeping in touch with ORNL. In late 1966, he was asked by the Beckman management to prepare a project report to examine the feasibility of developing a commercial product based on ORNL design. Using inputs from marketing and manufacturing divisions, Mr. Jacobsen prepared estimates regarding development costs, returns on investment, and projected annual gross profits. Mr. Jacobsen's proposal aiming "to improve the performance of ORNL design and to reduce the manufacturing cost" was approved by the management and work was started on this project in early 1967.

During a development period extending through about one and a half years Mr. Jacobsen had to solve a number of problems to make the rotor more efficient and less susceptible to operator error. One of the problems he had to solve related to the design of a suitable cap for the rotor. As mentioned earlier the rotor was to be loaded and unloaded while running. Obviously such a system would require some arrangement whereby the density gradient as well as the sample could be introduced and removed from the running rotor. A dynamic seal was used to solve this problem in the ORNL as well as the Beckman design. When mounted on the top of the rotor core, the lower part of the seal rotated with the rotor while the upper part, to which the external feed lines were connected, remained stationary.

It was found that to avoid rapid deterioration of the dynamic seal due to excessive heating between the rotating and the stationary parts of the seal, the rotor could only be run at low speeds (around 3,000 RPM) during the time the seal was mounted on the rotor. On the other hand, it was necessary to keep the inlet and outlet holes in the rotor core closed during the full speed run to eliminate the evaporation of the liquids to the compartment in which the rotor was running. (At 50,000 PRM the rotor surface runs at speeds reaching sonic velocity; to reduce the frictional drag, the compartment in which the rotor runs is evacuated to pressures of the order of a few microns. Any evaporation from the rotor would result in increased compartment pressure and consequently higher drag and frictional heating.) The problem of sealing the feed holes during the full speed run was solved by designing a cap which was used to replace the seal after the liquids had been fed in or taken out of the rotor. This cap rotated with the rotor after it was attached to it. Since there were no stationary parts in the cap, there was practically no frictional heating of the cap.

The ORNL solution to this capping problem is shown in Exhibit A-5. The cap is mounted on the rotor by holding the bearing housing stationary and pressing it down on the core projection. Sealing against evaporation to the rotor chamber is provided by the O-ring between the core projection and the vacuum cap. It is to be observed that due to the pressure difference across the cap there would be a force acting on the cap trying to push it upwards.* This upward force had to be resisted by the frictional force provided by the O-ring between the core projection and the vacuum cap. This necessitated the use of an extremely tight O-ring fit which in turn resulted in troubles during mounting and removal of the cap from the rotor running at 3,000 RPM. These operations took considerable time

*In Mr. Jacobsen's estimates the average pressure on the lower face of the vacuum cap can be up to 20 psi at full speed run. However, the pressure differential during the mounting or removal of the cap is usually negligible because of the absence of vacuum in the rotor chamber compartment and the low speed of the rotor.

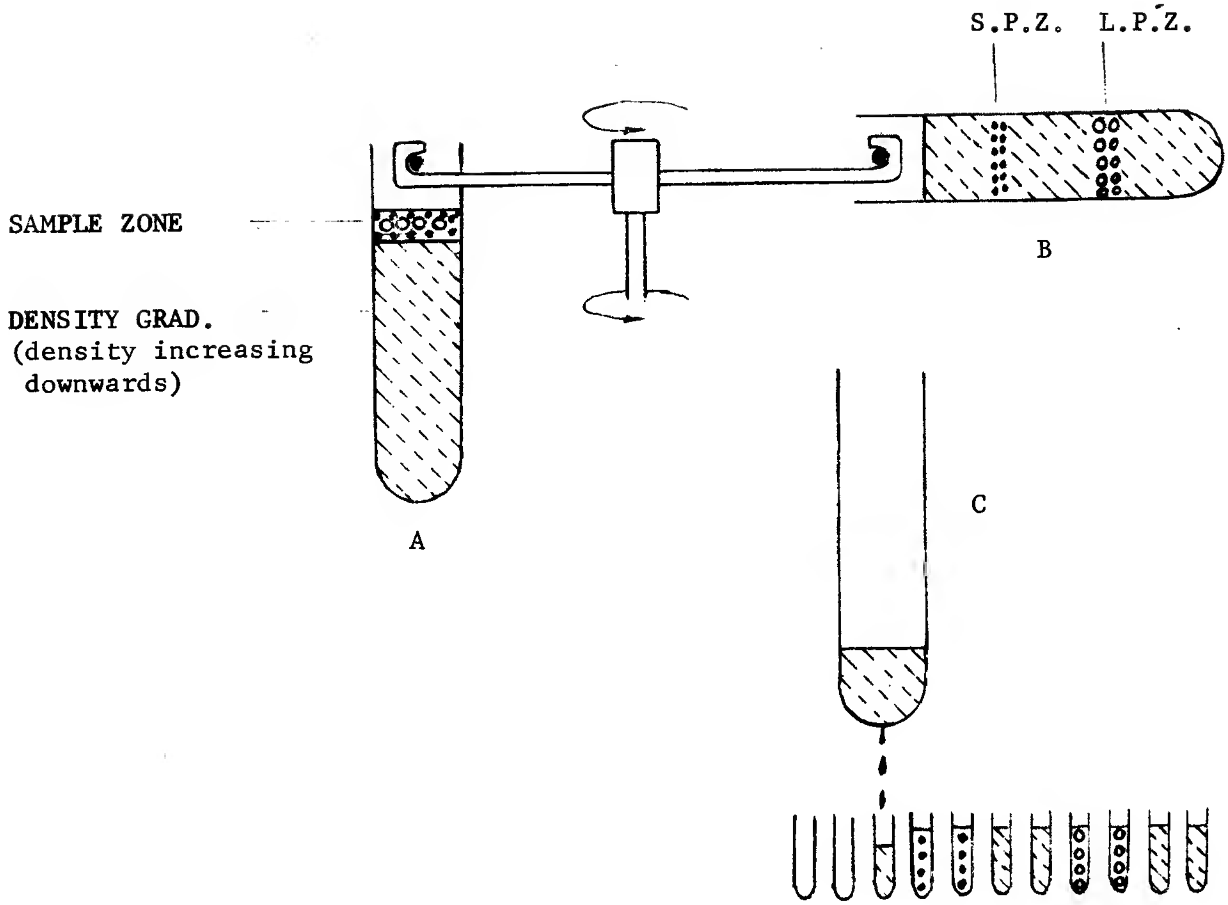
and often the cap-bearing housing would get very hot causing more trouble. Also, the large force necessary to remove the cap occasionally lifted the rotor itself from the drive head (not shown in exhibits). In fact, the ORNL design had to incorporate stops fixed to the rotor chamber compartment to prevent the rotor from lifting 'too much'. In Mr. Jacobsen's words, "The ORNL cap was good enough for a prototype coming from a research lab but left much to be desired for a commercial product. We needed a solution which would do the same job but in a simple and fool-proof manner." In his search for an appropriate solution Mr. Jacobsen had also to contend with the following limitations:

1. The space was very limited and the cap had to fit in the core projections designed for the liquid feeding seal. A view of the cap seat is shown in Exhibit A-6.

2. While undoubtedly the capping mechanism was important for the rotor operation, it was only a minor part in the overall rotor design. Consequently Mr. Jacobsen felt that the complete design of the cap should not take more than a couple of man-months.

3. From the marketing forecasts Mr. Jacobsen estimated the annual requirement for the capping mechanisms would be around 500. So he was interested in a solution suitable for this sort of production rate .

Note: Part B of this case describes Mr. Jacobsen's solution to this problem.



The gradient and the sample layer are introduced at rest (A). The tubes are accelerated to a horizontal position (B) and centrifuged until the desired separation is effected. The rotor is decelerated to rest, at which time the gradient and the separated zones (small particle zone, S.P.Z.; large particle zone, L.P.Z.) are recovered (C), usually through a small hole punctured in the bottom of the tube.

Note that the location of the separated zones in this case does not coresspond to their equilibrium position which would be the case if the separation was done on the basis of density differences only.

EXHIBIT A-1: SCHEMATIC OF CENTRIFUGAL SEPARATION BASED UPON DIFFERENCES IN SEDIMENTATION RATES.

The award-winning design of the Model L2-50 matches its elegant capabilities. It is a compact, operator-oriented instrument. The control panel is conveniently organized, height to the working surface is a comfortable 36 inches, and easy access to major components is provided by three front swing-out panels.

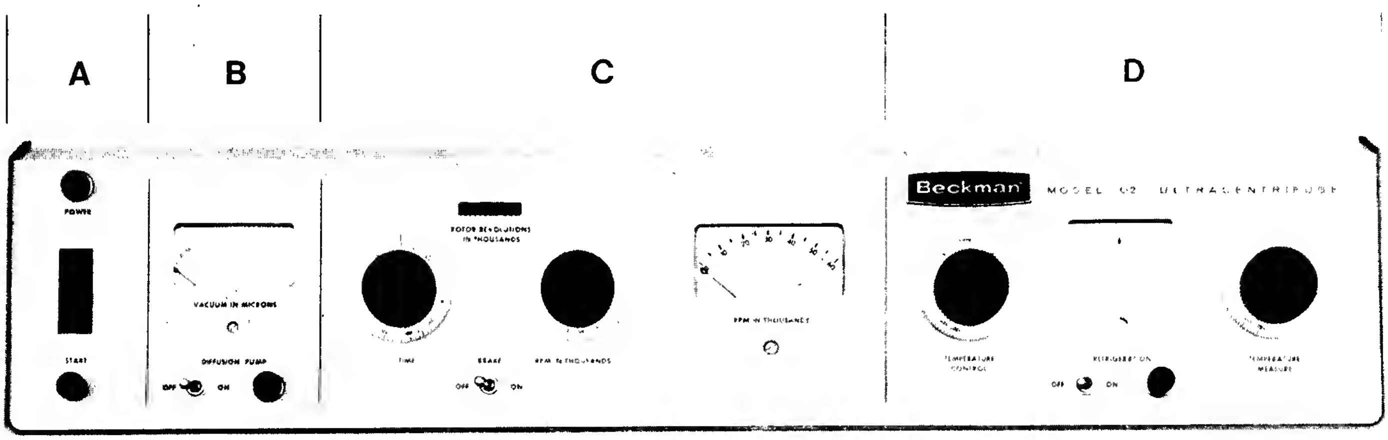
Panel Controls Follow the Sequence of Operating Steps

A Main power switch is in the first section, with pilot light above it, start button below.

B Next is the gauge indicating vacuum in the rotor chamber. If the optional diffusion pump is included (Model L2-HV), an on/off switch for the pump and a pilot light are provided below the vacuum gauge.

C Center section of the control panel houses time and speed controls. Clock dial at left can be set to turn off the rotor drive at any time up to 5 hours. Desired rotor speed is set on the second black dial; the tachometer beside it indicates running rpm. An electromagnetic totalizer-counter shows true accumulated revolutions and hence can serve as an accurate check on rotor speeds. Slow or fast braking is provided by the switch below.

D Temperature controls and indicators are grouped together in the final section. Dial at left is set to the degrees Centigrade required; a thermistor-controlled regulating system closely monitors temperature directly in the hollow-stem rotor to maintain selected level. Temperature is read out on dial at far right, by means of a central null meter; below, the refrigeration switch and its pilot light.



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Other Features of the Model L2-50

E *Rotor Stabilizing arm* smooths rotor operation during acceleration and deceleration, helping to prevent the remixing of delicate separations. A removable shield attached to the stabilizer forms a barrier to external radiant heat.

F *Large rotor chamber* accepts two high-capacity rotors, the Type 19 (1,500 ml) and the SW 25.2 (180 ml in 3 swinging buckets), as well as other high-volume fixed-angle and swinging-bucket rotors. Chamber is surrounded by two inches of forged steel armorplating that protects both instrument and equipment.

G *Central control knob* does double duty; it opens and shuts the rotor chamber cover and contains the vacuum pump switch.

H *Oil supply* is immediately visible; rotor drive cuts off if oil reaches minimum operating level.

I *Refrigeration system* holds rotor at any preselected temperature from 5° below ambient to 0°C. To minimize condensation in the chamber, the system cannot be turned on until the vacuum pump is operating.

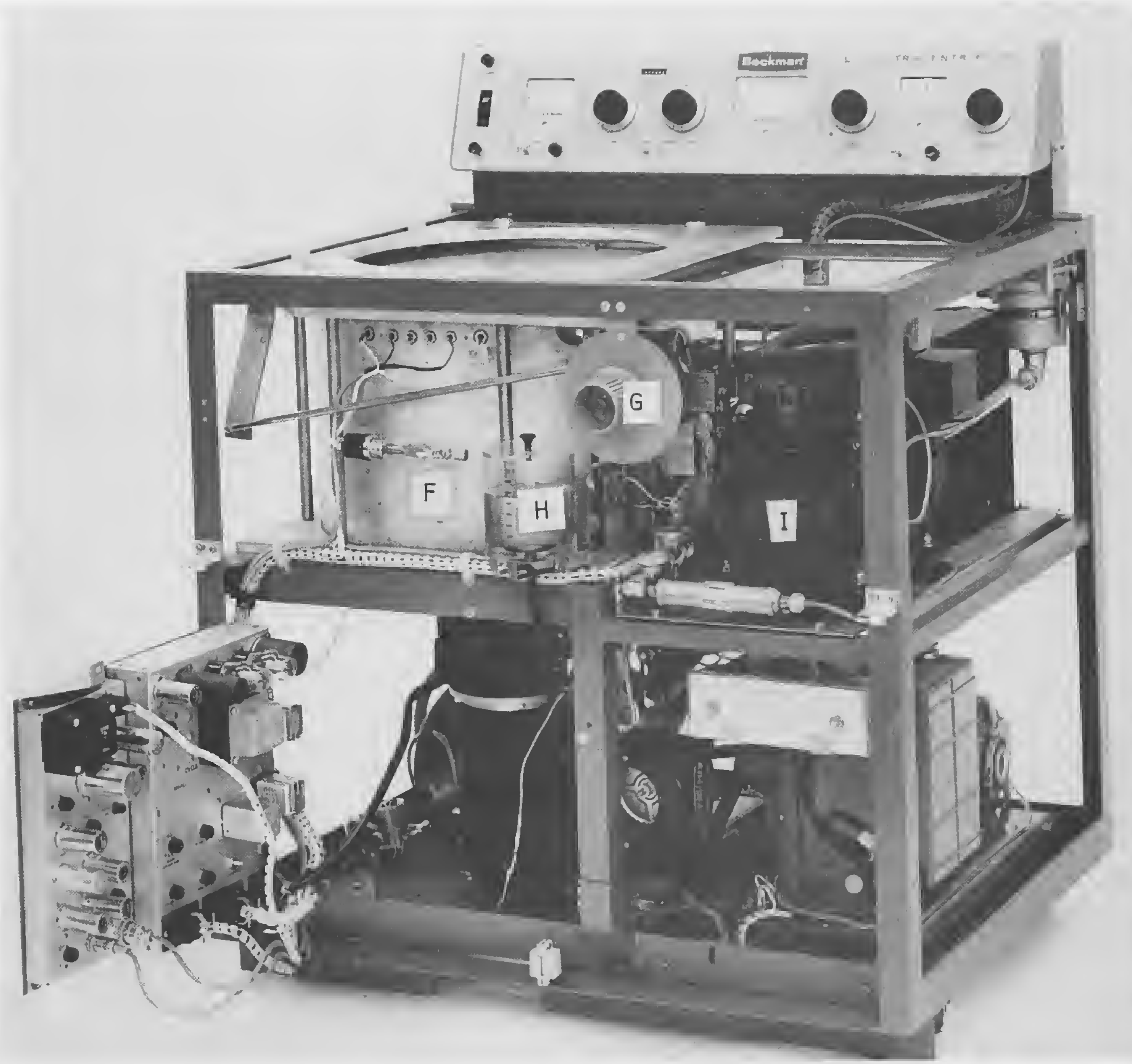
J *Geared electric drive* operates routinely at 50,000 rpm for 1.2 billion revolutions or more; drive replacement is easily accomplished by a Beckman field engineer.

K *Automatic electronic monitor* carries out preset speed, temperature, and braking instructions, includes fail-safe circuitry that safeguards operation, and is virtually unaffected by line-voltage fluctuations.

L *Heavy-duty vacuum pump* is mounted on slides for easy access; it lowers rotor chamber air pressure to well under 100 microns. At option, the instrument comes with oil diffusion pump, necessary water connections, and additional controls which can provide a vacuum to below 5 microns. If originally ordered, this high-vacuum capability changes the instrument's Model designation to L2-HV; it can be added at any later date by factory-trained field engineers.

Space-Saving Installation

Design of the L2-50 provides easy access to its major components. In fact, all normal maintenance can be done from the top or front panels. This helps to save laboratory space, because the instrument can be installed side-by-side with other equipment.

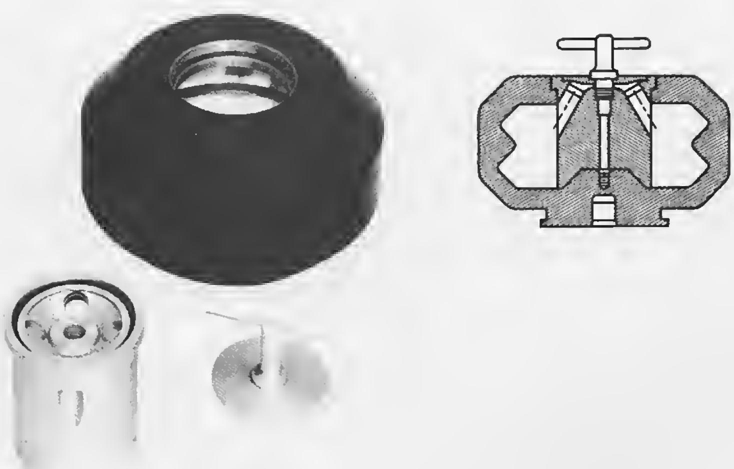


A. <u>FIXED ANGLE ROTOR</u>

Total Capacity	108 ml.
Max. Rated Speed	65000 rpm.
Max. Force	368000 g.

B. <u>SWINGING BUCKET ROTOR</u>

Total Capacity	180 ml.
Max. Rated Speed	25000 rpm.
Max. Force	106900 g.



C. <u>BATCH TYPE ROTOR</u>

Total Capacity	1600 ml.
Max. Rated Speed	16000 rpm.
Max. Force	27500 g.

EXHIBIT A-3: THREE TYPICAL ULTRACENTRIFUGE ROTORS

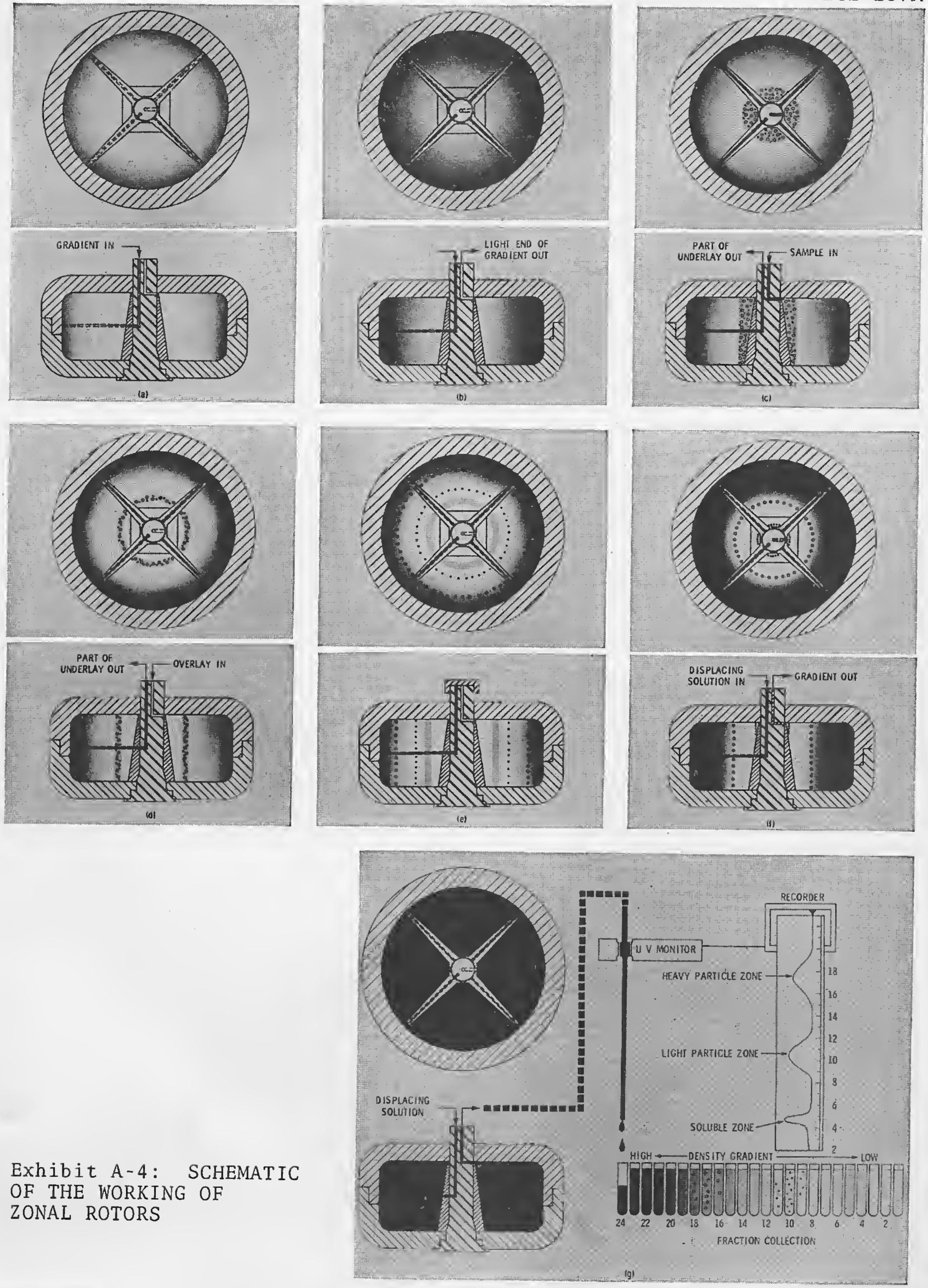
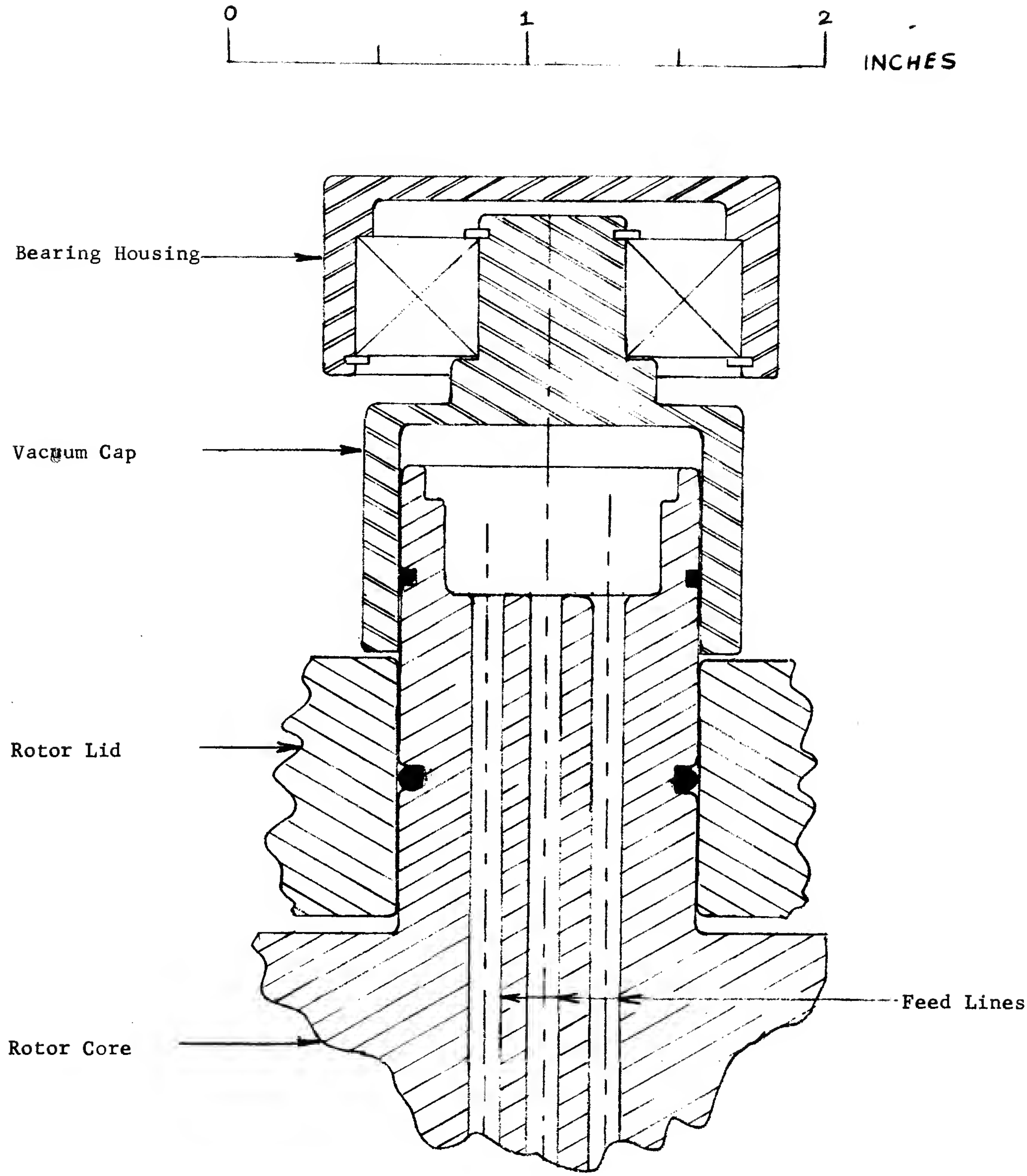
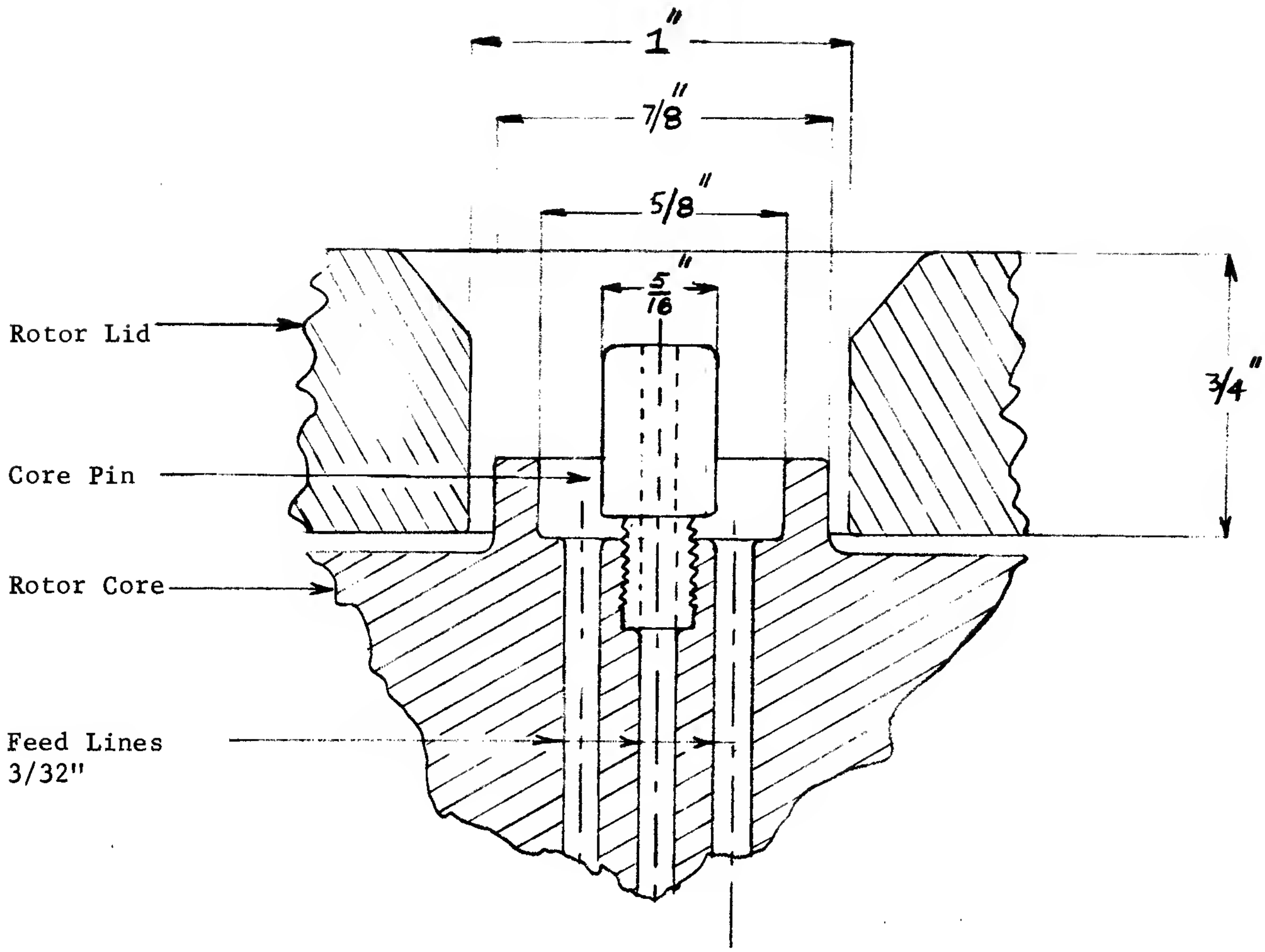


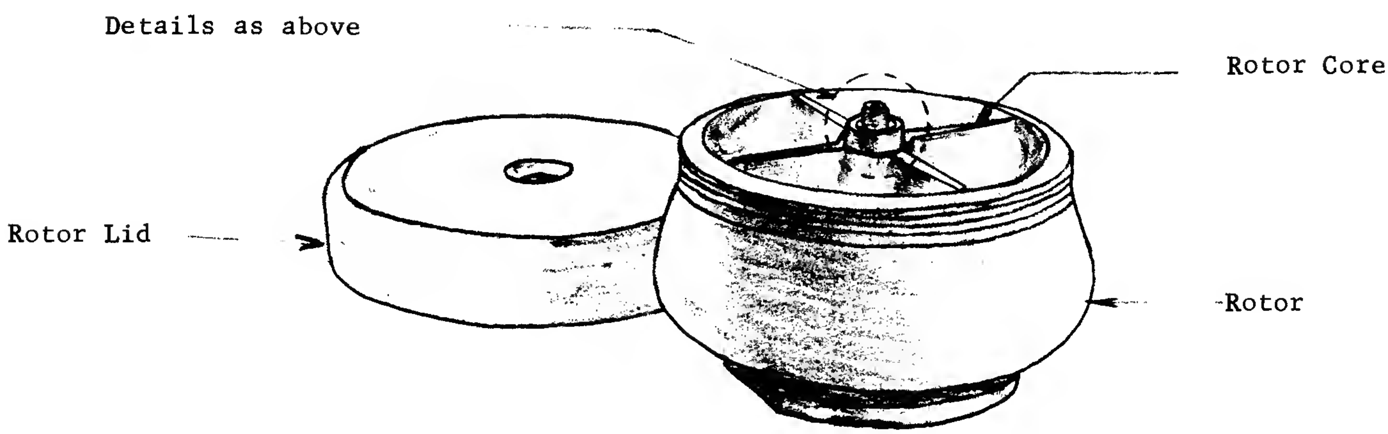
Exhibit A-4: SCHEMATIC OF THE WORKING OF ZONAL ROTORS



Note : Some of the details not related to this problem have been omitted
or modified in the above drawing.

EXHIBIT A-5: A VIEW OF THE ORNL CAP





Note: Some of the details not pertinent to this problem have been omitted from the above drawing.

EXHIBIT A-6: A VIEW OF THE CAP SEAT FOR THE BECKMAN ROTOR

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DESIGN OF A CENTRIFUGE ROTOR CAP (B)

Mr. Jacobsen's final solution to the rotor cap problem is shown in Exhibit B-1. His solution incorporates a hollow, slotted cone mounted on the pin projecting out of the core. Also, the vacuum cap has a cylindrical hole in which the depressor cup moves axially, actuated by the up and down motion of the push button. The essential difference between this solution and the ORNL solution is the manner in which they counter the upward force acting on the cap. In Mr. Jacobsen's design this is achieved by creating an interference between the cone and the lower projection of the vacuum cap (see details in Exhibit B-1). When the cap is pressed down on the core projection, the cone contracts enough to enter the hollow cylindrical hole in the vacuum cap. During removal the cone is released by the downward motion of the depressor cup. In Mr. Jacobsen's design the O-rings don't have to resist any upward force and they don't have to have very close fit; consequently the cap is mounted or removed almost effortlessly.

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"I got the idea of using a slotted cone from the similarity of the problem to the one in Capstan lathes where a collet type action is used to grip the bar stock. However I had to go through a number of variations before arriving at the final solution," recollects Mr. Jacobsen. Among the variations he attempted was the use of a screw to get the axial motion which is obtained by the push button in the final design. The choice of a proper material and configuration for the cone proved to be a rather difficult problem. The problem here was to have a cone which wouldn't be very stiff yet would be strong enough to withstand the compressions during mounting and removal without suffering a permanent set. His early attempts were to try conventional metals such as stainless steel and titanium with different slot configurations; none of them worked. Subsequently a number of plastics such as Delrin and Polypropylene were tried. The final material for the cone is Noryl, a high strength plastic which also is highly corrosion resistant. As would be expected, the final design involves very close tolerances.

The present solution has been extensively tested and found to be trouble free for long duration runs. None of the units sold in the market has had a complaint recorded so far, and Mr. Jacobsen is confident that the units will continue to prove satisfactory.

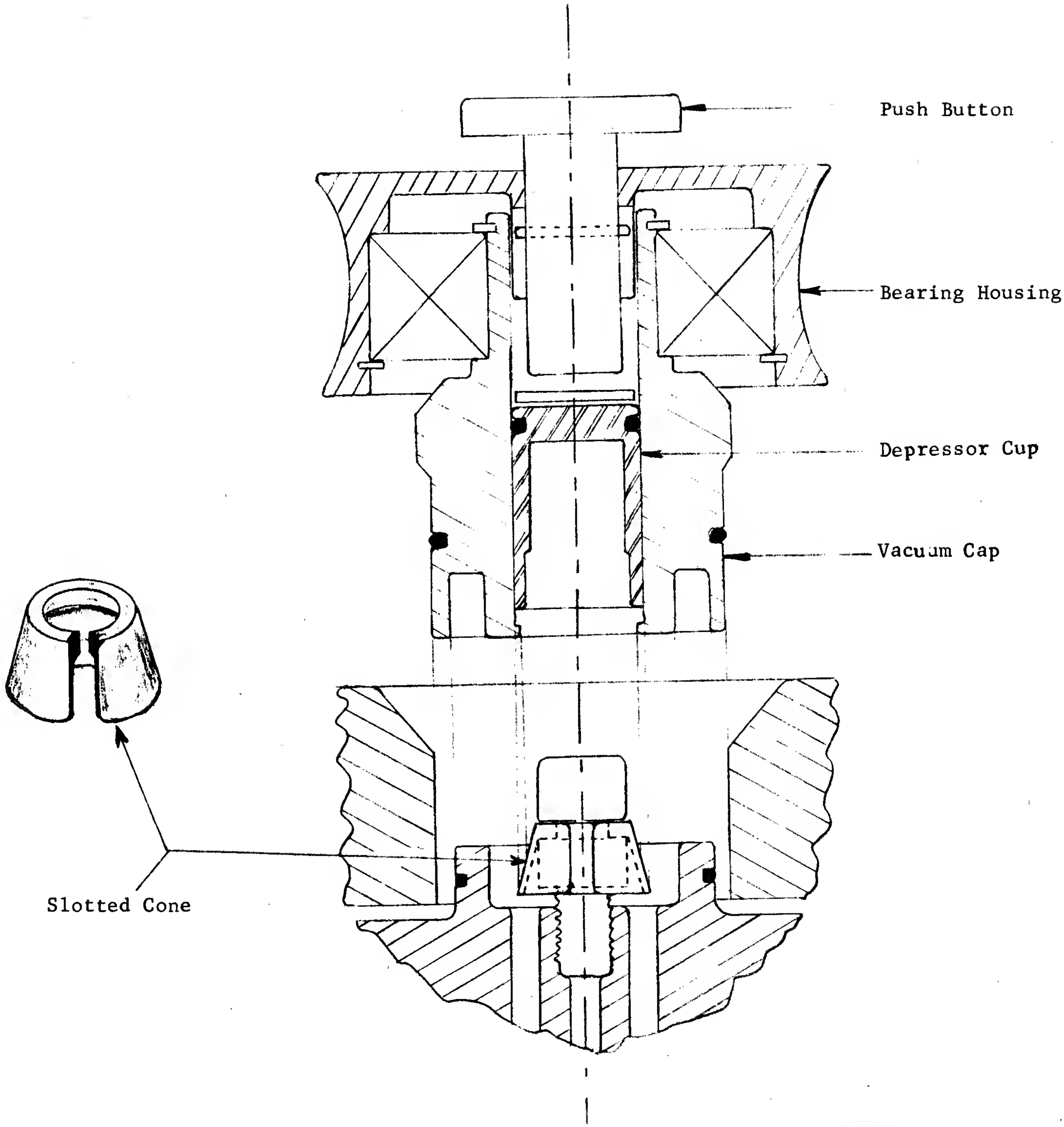

EXHIBIT B-1: A VIEW OF MR. JACOBSEN'S SOLUTION FOR ROTOR CAP